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Simple and Scalable Electrochemical Synthesis of 2,1-Benzisoxazoles and Quinoline N-Oxides

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Cathodic reduction of the nitro moiety and subsequent intramolecular cyclization affords different substituted 2,1benzisoxazoles and quinoline N-oxides. This methodology allows the synthesis of two different type of heterocycles from common simple starting materials, using electrons as a sole reagent for this transformation. The electrolysis can be conducted in a very simple undivided electrolysis cell under constant current conditions. This permits working on larger scale compared to other electrochemical methodologies and represents a significant advantage.

Nitrogen- and oxygen-containing heterocycles represent a wide branch of research in organic chemistry.¹ In particular, 2,1benzisoxazoles are important structures in both medicinal and synthetic chemistry. Several derivatives exhibit antibacterial properties,² whereas others turned out to be useful for the treatment of nervous diseases.³ Recently they have arisen as precursors of important motifs in metal catalysis, especially with Rh,⁴ Au,⁵ or other metals.⁶ Another significant type of heterocycles with an N-O bond are quinoline N-oxides. In analogy to 2,1-benzisoxazoles, they are important motifs and precursors in medicinal chemistry,⁷ but due to their 1,2-dipolar N-O bond, they are also very versatile intermediates in organic synthesis.⁸ 2,1-Benzisoxazoles have been usually synthesized starting from nitro derivatives using Sn, Sn salts,⁹ or with other metals as reducing agents such as Zn¹⁰ or In.¹¹ Recently, Corma has introduced Pt-supported nanoparticles in combination with hydrogen as an alternative methodology.¹² In this sense, electro-organic synthesis has experienced a renaissance during the past years,¹³ as the substitution of chemical reagents by electricity entails a step forward in terms of green chemistry.¹⁴ The electrochemical formation of 2,1-benzisoxazoles was reported, but due to many drawbacks being far from synthetic exploitation. Several studies with o-nitrobenzaldehyde or onitroacetophenone using Hg cathodes tracing intermediates and potential products but without any synthetic application.¹⁵ Then Kim and co-workers developed a general approach employing a sacrificial lead cathode.¹⁶ Recently, Peters showed that stable electrodes could also yield the 2,1-benzisoxazoles.¹⁷ However, in all cases a divided cell under potentiostatic conditions was used, representing the maximum effort that impedes the use in scalable syntheses. This is effectively circumvented if galvanostatic conditions and an undivided cell are employed. This simplifies the practical aspect and, consequently, makes it easier and suitable for synthetic applications. Indeed, no evidence of large scale has been given in the previous reports.^{16,17} In the work of Peters, the electrolysis was also performed in the presence of chlorophenol as additive in large excess (10 times the starting material).

Moreover, when galvanostatic mode was tried, a mixture of products was obtained, proving the difficulties of the transformation under these conditions, even employing a divided cell and working at extremely low current densities.¹⁷ Finally, it is worth mentioning that no strong EWG groups were present in the previous scopes.



Scheme 1. Electrochemical approach: Single methodology for the synthesis of both 2,1-benzisoxazoles and quinoline *N*-oxides from a common starting point.

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Quinoline N-oxides are mostly obtained by oxidation of the corresponding quinoline.8 The use of nitroso derivatives in combination with diazoesters and metal catalysis has also been reported.¹⁸ Another feasible approach would be the cyclization starting from 2-nitrocinnamaldehydes. Nevertheless, when this reduction is carried out by classical methods, the obtained product is the quinoline, and not the N-oxide.¹⁹ To our knowledge, there is only one general methodology in which the quinoline N-oxide is obtained from the corresponding cinnamaldehyde: through the use of bakers' yeast,²⁰ with some structural limitations and careful control of the amount of NaOH employed, since a subsequent reduction to the guinoline can occur easily, affording a mixture of products. With our recent experience in the electrochemical reduction of aryl nitro derivatives,²¹ nitrones²² and synthesis of N-containing heterocycles,²³ we envisioned a practical electrochemical access to both, 2,1-benzisoxazoles and quinoline N-oxides starting from 2-nitrobenzaldehydes. A strategy which allows the synthesis of two different heterocycles from a common starting point, using electrons as reagent, in a time-saving, atomeconomy and sustainable way (Scheme 1). The whole approach would make the experimental conditions for the formation of the 2,1-benzoisoxazoles far more simple, while it would also expand the possibilities with the formation of the quinoline Noxides.

First, we carried out the reaction optimization for the 2,1benzisoxazoles. The results are summarized in Table 1. The initial experiments were based on our previous work.²¹ We used o-nitrobenzaldehyde as test substrate, in an equal volume of EtOH and H₂O, a charge of 4.0 F, with glassy carbon and borondoped diamond (BDD) as anode and cathode respectively. First, the current density was varied. A value of 2.4 mA/cm² (Entry 3) was found to be the optimal one, offering the best balance between yield and reaction time. However, the solvent turned out to be a more sensitive parameter. Upon testing various solvent mixtures, protic solvents proved vital to ensure higher yields. Alcohols (Entries 5 and 6) gave better yields than aprotic solvents (Entries 7-9). HFIP, a solvent that has shown excellent performance in electrochemical reactions,²⁴ allowed us to raise the yield up to 53%. In addition, HFIP is mildly acidic,²⁵ a role performed by toxic chlorophenol in the previous work of Peters¹⁷ that demonstrated to be crucial. Therefore, the employment of HFIP also avoids the use of additives and it can be easily recovered and recycled after work-up. These facts makes the whole electrolysis simpler and more practical. Addition of 4 vol% of acetone facilitated the dissolution of the substrate within the electrolyte. The use of other solvent proportions, acidic solvents or the absence of any of them resulted in no improvement (see ESI). Further tests focused to the applied charge employed. Less applied charge than the theoretical amount led to a logical decrease of the yield (Entry 11), whereas an increase of applied charge had the same effect (Entries 12-13). The latter might be explained by the fact that the N-O bond can be subsequently over-reduced, vanishing the product and affording the corresponding aminobenzaldehyde. Finally, some experiments with metallic cathodes revealed no improvement over BDD cathodes (Entries 14-15).

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Table 1. Optimization of the electrochemical synthesisticat ວິມູ່ມະ benzisoxazol 2a under constant current conditions.1039/C9CC06054E

NO ₂ O H –		glassy carbon cathode current density, applied charge solvent, NBu ₄ BF ₄ [0.01M], rt		► N-0 H	
Entry ^a	Current density (mA/cm²)	Applied charge (F)	Solvent (1:1) ^b	Cathode	Yield (%)⁴
1	6.5	4	$EtOH:H_2O$	BDD	15
2	4.1	4	EtOH:H ₂ O	BDD	23
3	2.4	4	EtOH:H ₂ O	BDD	25
4	0.6	4	EtOH:H ₂ O	BDD	26
5	2.4	4	MeOH:H ₂ O	BDD	31
6	2.4	4	iPrOH:H ₂ O	BDD	21
7	2.4	4	$CH_3CN:H_2O$	BDD	<5
8	2.4	4	DMF:H ₂ O	BDD	17
9	2.4	4	THF:H ₂ O	BDD	19
10	2.4	4	$HFIP:H_2O^c$	BDD	53 ^e
11	2.4	3	HFIP:H₂O	BDD	23
12	2.4	5	HFIP:H₂O	BDD	51
13	2.4	6	HFIP:H₂O	BDD	42
14	2.4	4	HFIP:H₂O	Pt	41
15	2.4	4	HFIP:H₂O	Leaded bronze	26
16	6.5	4	EtOH:H ₂ O	BDD	15

^a Concentration of o-nitrobenzaldehyde was 20 mM. ^b 4% vol of acetone was used, unless otherwise is noted. ^c Without acetone, yield dropped down to 14%. ^d Yields were determined by NMR, using 1,3,5-trimethoxybenzene as internal standard. ^e Isolated yield = 42%.

The scope of the reaction was then explored. The results are displayed in Scheme 2. A naphthyl group (**2b**) and a strong activated ring (**2c**) afforded the 2,1-benzisoxazoles in good yield. The introduction of strong electron-withdrawing groups attached to the aryl (**2d-f**) entails a novelty, since the previous scopes did not include any example with strong EWG.^{16,17} The tolerance of carbon-halogen bonds (**2g** and **2h**), often very sensitive to electrochemical conditions, was also achieved.



^a 4% vol of acetone was used. ^b large scale: 12 mmol of **1c**, yield was 55%. ^c Q = 7F.

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In order to demonstrate the scalability of the process we converted 12 mmol of reactant **1c** to product **2c**. The yield was similar to the one obtained when the reaction was run on a smaller scale (0.5 mmol).

Subsequently, we focused onto the vinylogous conversion and to establish suitable electrolysis conditions for the synthesis of quinoline *N*-oxide **4a** starting from 2-nitrocinnamaldehyde **3a**. Starting point for the electrosynthetic screening (Table 2) were the electrolytic conditions for the generation of 2,1-benzoisoxazole **2a**.

Table 2. Optimization of the electrochemical synthesis of quinolineN-oxide 4a under constant current conditions.



^{*a*} Concentration of o-nitrocinnamaldehyde was 20 mM. ^{*b*} 4%vol of acetone was used. ^{*c*} Yields were determined by NMR, using 1,3,5-trimethoxybenzene as internal standard. ^{*d*} Isolated yield. ^{*e*} Same yield was obtained using NEt₄BF₄ as supporting electrolyte.

Initial tests varying current densities (Entries 1-4) revealed that the previous current density of 2.4 mA/cm² still provided the highest yields (Entry 3). However, using the theoretical amount of applied charge (4 F) conversions were low. This fact can be explained by the less-favoured condensation between the hydroxylamine and the carbonyl moiety of the enal, due to the conjugation with the alkene. If the condensation is not fast, a reoxidation of the hydroxylamine to the nitroso species may occur, something which can also led to secondary products, such as azoxybenzenes.²⁶ We increased the amount of applied charge (Entries 5-7) to 6 F to obtain nearly full conversion (entry 6) and higher yields. Further screening on solvents used (Entries 8-9) indicate that the initial mixture of HFIP:H₂O (1:1) was still superior to other media. Upon testing the cathode materials, vitreous carbon and leaded bronze as electrode materials did not provide a significant increase in yields. However, platinum foil (Entry 10) as cathode improved the yield slightly, so it was chosen over BDD. We found a final issue regarding the supporting electrolyte. Because of the Very MgA/polanty of quinoline *N*-oxides, we could not isolate **4a** as a pure compound, since it always co-eluted with the supporting electrolyte upon purification via silica gel chromatography. This was solved by replacing the cation of the supporting electrolyte, using NEt₄⁺ instead of NBu₄⁺, which is much more hydrophilic and can be removed with a simple aqueous extraction, providing the same yield.

The scope for synthesis of quinoline N-oxides 4 was then explored and it is displayed in Scheme 3. All the 2nitrocinnamaldehydes 3 were easily accessible by a classical reaction,27 Wittig olefination using commercial (triphenylphosphoranylidene)acetaldehyde. The presence of EWG and EDG did not affect the yields noticeably, and electrochemically sensitive groups, such as CN (4e) and halogens (4g and 4h) were again tolerated and did not lower the yields significantly. The scaling-up was performed with 5 mmol of compound **3a**, with a similar yield for **4a** in comparison to the small-scale reaction, although a little increase in the amount of charge was necessary. These quinoline N-oxides can be derivatized to the corresponding quinolines employing electrochemical conditions.28





^a 4% vol of acetone was used. ^b 5 mmol of 3a, yield was 38% (6.5 F)

In summary, we established an electrochemical protocol for the synthesis of different substituted 2,1-benzisoxazoles and quinoline *N*-oxides starting from 2-nitrobenzaldehydes, using just electric current as reducing agent, expanding the electrochemical possibilities in the synthesis of different heterocycles. The utilization of an undivided cell under constant current conditions represents the key advantage, since it offers a simpler and easier way to conduct the electrolysis and permits also working at larger scales in comparison with other electrochemical methods described up till now. Strong EWG were included for the first time and a large variety of

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substitution patterns and functional groups are tolerated, even those which are sensitive to electrochemical conditions.

Conflicts of interest

There are no conflicts to declare.

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